Opportunities for X-ray Absorption Spectroscopy at NSLS-II

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Ceramics Division
National Institute of Standards and Technology

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My mandate from Tony

- New scientific directions and technical innovations in molecular environmental science and geochemistry XAS studies
- Instrumentation, optics, and computing trends
- New technologies and scientific possibilities for XAS at low concentration and small spot size

Fortunately he's given me 20 whole minutes to talk!

NSLS-II Sources and their XAS applications

Damping wiggler Highest flux for low concentrations, low detector efficiency, or pump-probe dynamics. $\sim 1\,\mu$ microprobe. Resolution refining monochromator. Somewhat quick XAS. 5.5–90 keV.

DW side station Very high flux, unfocused tender x-rays *(X15b/X19a on steroids)*

3 pole wiggler Conventional XAS (4–25 keV) *(X11a)*. Quick XAS *(X18b)*. $< 5\,\mu$ microprobe *(X26a)*.

Soft bend Low energy (100–1000 eV) XAS *(U7a)*. Medium energy (1–6 keV) x-ray bulk XAS *(X15b/X19a)*. Medium energy (1–6 keV) x-ray microprobe.

Undulator Smallest spot size with excellent flux, XAS by scanning the gap.

Hutch instrumentation

Essential to the E&ES community

- In-line controlled atmosphere chamber
- High-volume, high-throughput cryostats

Other things to consider

- Automated gas handling: ion chambers and/or hazardous gases
- Liquid delivery: water, LN
- Ample power and signal capacity
- Laboratory support

Detector technology

- Energy discriminating (Ge or Si-drift) will be the work-horse
- Wavelength dispersive (multilayer analyzers, bent Laue, etc) will fill many important niches
- High-rate and large number detectors from the instrumentation division

Data acquisition and data analysis software

Common DAQ infrastructure Data acquisition software should be as similar as possible on all beamlines. The DAQ solution should be extensible, open source and platform agnostic. A client/server framework (probably based on EPICS) is very attractive.

Beamline monitoring

Metadata

Analysis automation

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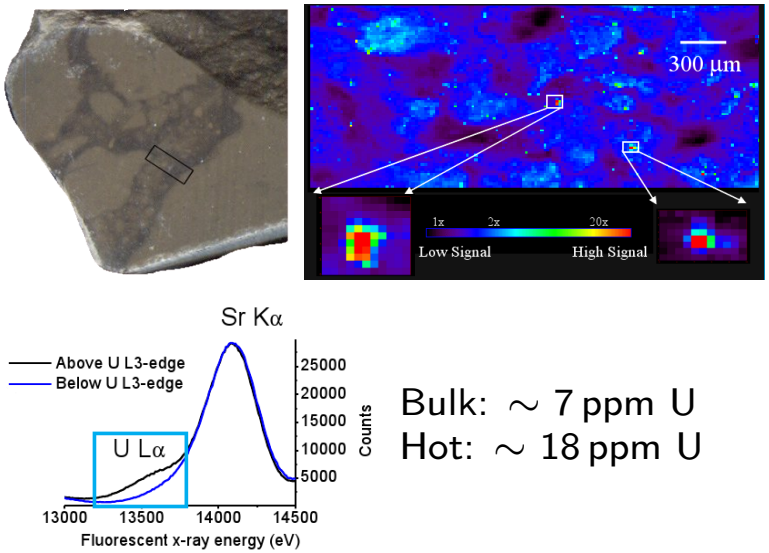
Analysis automation High-quality data processing and analysis needs to be better incorporated into the DAQ architecture. Raw XAS data should be presented to the user with baseline processing. Automated fitting available for high-throughput or screening applications.

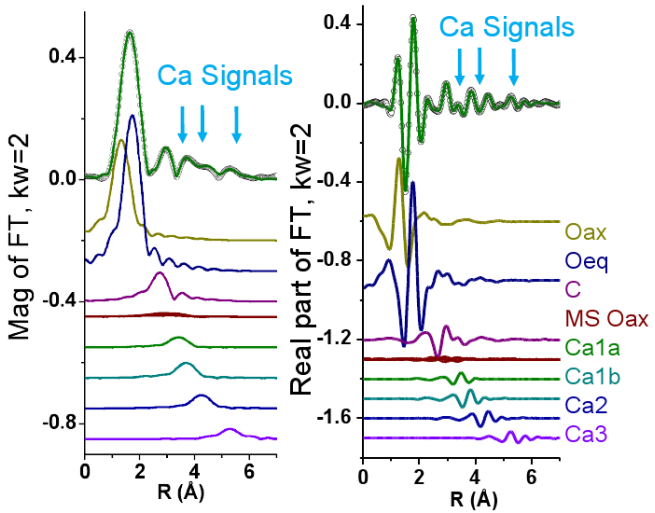
EXAFS at environmentally relevant concentrations



Bulk: $\sim$ 7 ppm U
Hot: $\sim$ 18 ppm U

Evidence for stable uranium incorporation in an ancient calcite – a plausible squestration strategy.

S.D. Kelly, E.T. Rasbury, S. Chattopardhyay, A.J. Kropf, K.M. Kemnner, *Evidence of a stable uranyl site in ancient organic-rich calcite.* Environ. Sci. Technol. (2006), 40(7), 2262-2268.



After 2.5 days of continuous data collection at an APS undulator beamline, high quality data was obtained an subject to detailed analysis.

APS 10ID	10^{11} ph/s	2.5 days
NSLS X26a		
NSLS-II DW		

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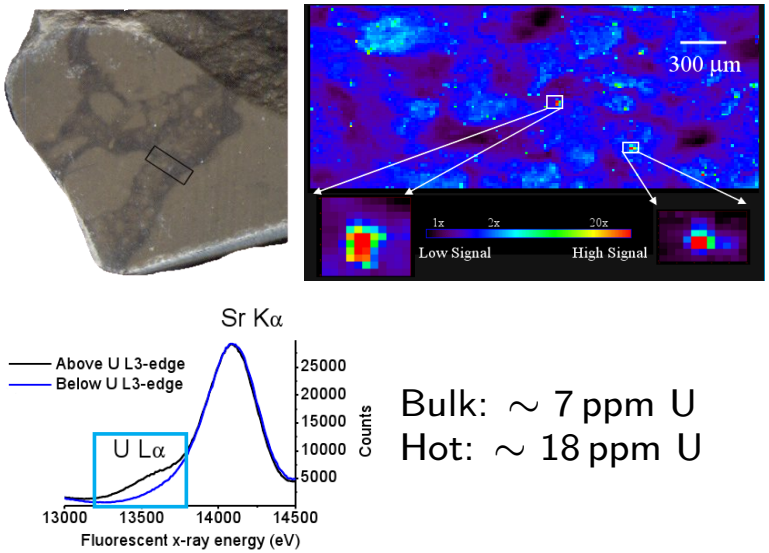

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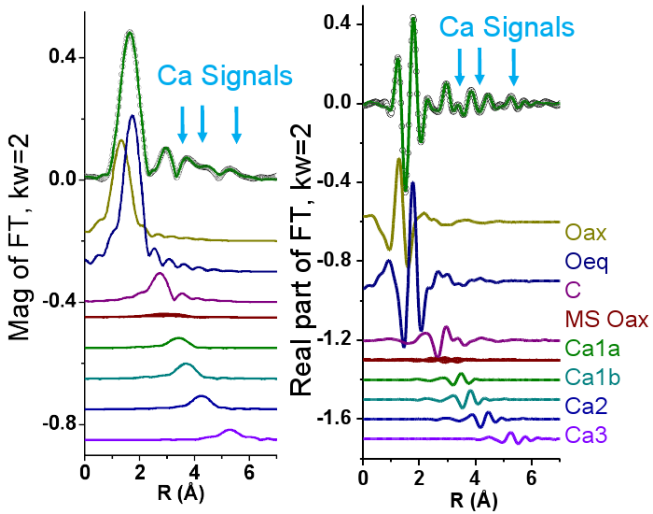

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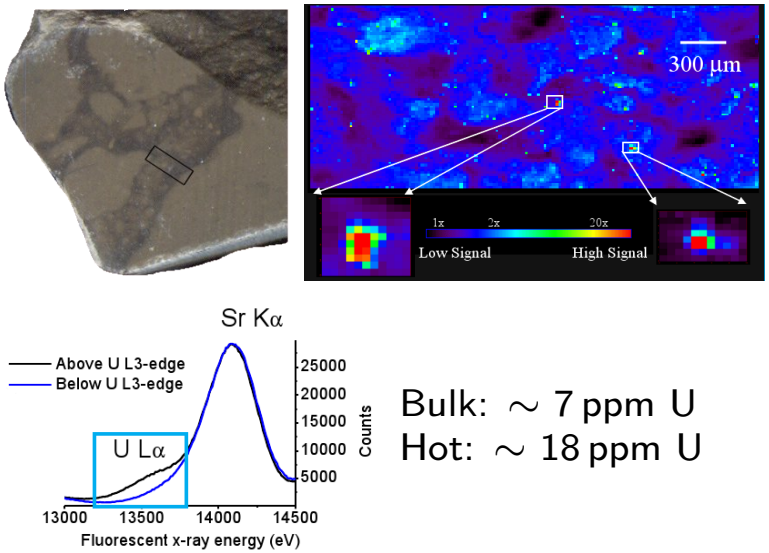

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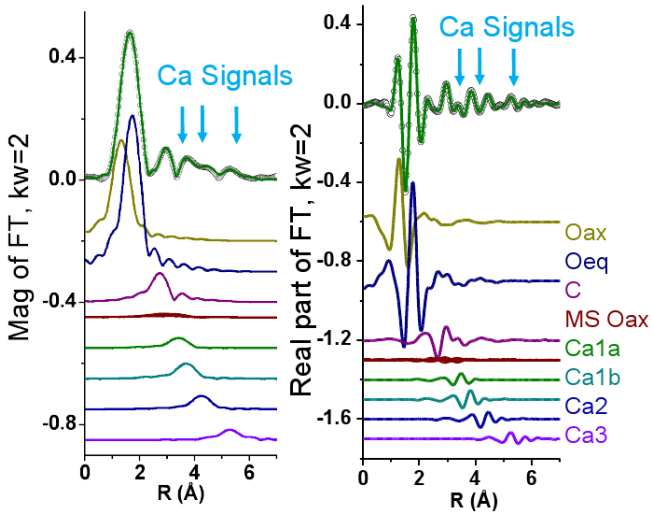

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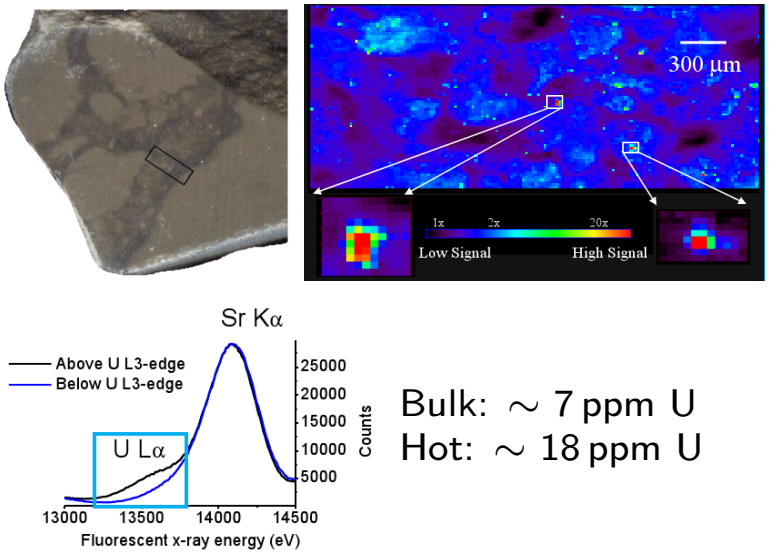

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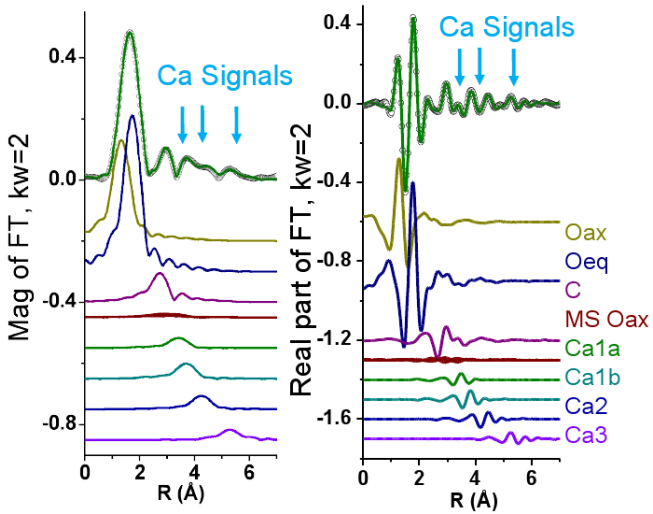

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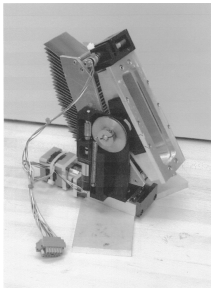
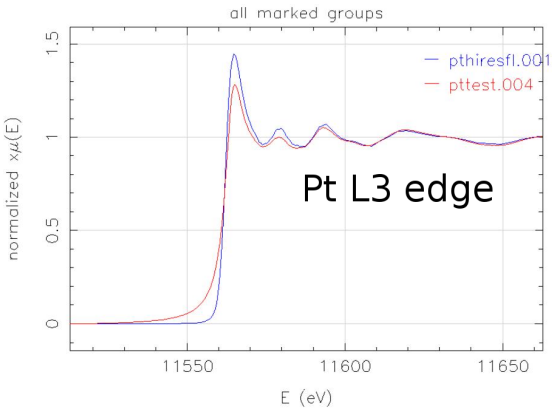
NSLS X26a	$3\text{x}10^{8}$ ph/s	24 months
NSLS-II DW	10^{12} ph/s	6 hours

High resolution fluorescence detection



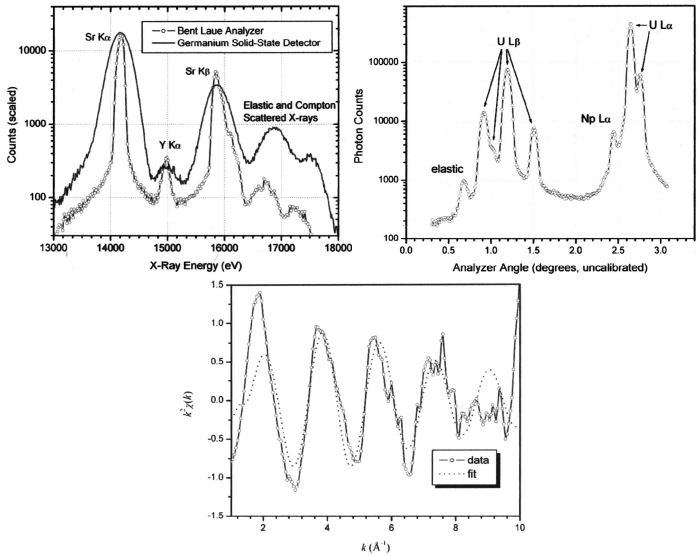
The bent Laue analyzer does wavelength dispersive detection of the fluoerescence signal.



Imagine doing U^{4+}/U^{6+} speciation with this level of resolution!

A. J. Kropf, R. J. Finch, J. A. Fortner, S. Aase, C. Karanfil, C. U. Segre, J. Terry, G. Bunker, and L. D. Chapman, *Bent silicon crystal in the Laue geometry to resolve x-ray fluorescence for x-ray absorption spectroscopy*, Rev. Sci. Instrum. (2003), 74, 4696-4702

1 part Np in 160 parts U_3O_8

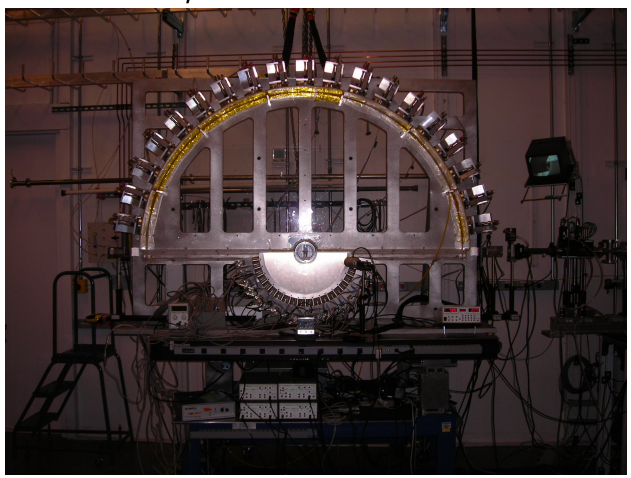


High-resolution EXAFS

With high flux, experiments with low detector efficiency are feasible.

LERIX: Soft XAS edges using hard x-rays

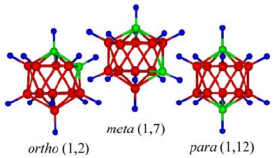
APS, Beamline 20ID

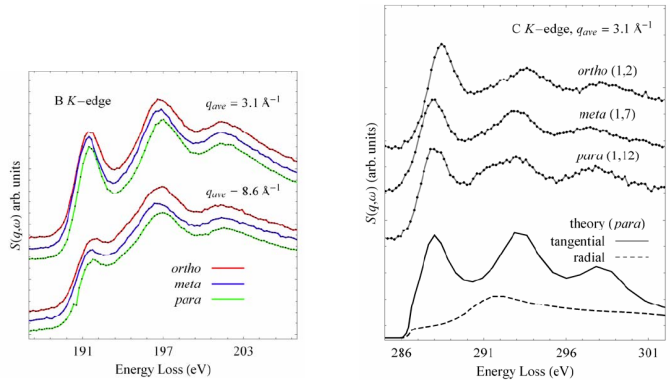


Incident energy: 10 keV

Near dipole conditions: 1750 and 320 cps at B and C K-edges on top of 2360 and 1250 cps

Three isomers of $C_2B_{10}H_{12}$





T.T. Fister, F.D. Vila, G.T. Seidler, L. Svec, J.C. Linehan, J.O. Cross, *The Local Electronic Structure of Dicarba-closo-dodecarboranes $C_2B_{10}H_{12}$*, J. Am. Chem. Soc. *accepted*

Too cool!

No vacuum, no special sample prep, hard x-rays in!

What *you* should be thinking about

- What novel experiments can **you** imagine which use the new sources NSLS-II will provide?
- What instrumentation or infrastructure would **you** like to see at beamlines at NSLS-II?

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A special note to the university faculty among us

NSLS-II (and other synchrotrons) need good staff scientists. Encourage some of your students to fall in love with synchrotron radiation.